Pine Chemicals Association, Inc.

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June 3, 2002

The Honorable Christine Todd Whitman
Administrator
U.S. Environmental Protection Agency
P.O. Box 1473
Merrifield, VA 22116

Attention: Chemical Right-to-Know Program

Re: Response to Comments and Amendments to Pine Chemicals Association, Inc. Test Plan for Rosin and Rosin Salts

Dear Ms. Whitman:

The Pine Chemicals Association, Inc. (PCA) HPV Task Force is pleased to submit its response to comments received on its September 2001 Test Plan for Rosin and Rosin Salts. We have carefully reviewed the comments submitted by the Environmental Protection Agency (EPA) in March 2002 and by the Physicians Committee for Responsible Medicine in February 2002. This document responds to those comments and amends our September 2001 Test Plan. We have organized the submission by subject matter in the same order as our Test Plan.

RESPONSE TO COMMENTS & AMENDMENTS TO TEST PLAN

Description of Rosin and Rosin Salts/Composition

EPA's comments noted that PCA did not provide typical compositional ranges in the Test Plan for the three types of rosin (CAS # 8050-09-7). The following table from the PCA (1987) publication "Tall Oil and its Uses-II" illustrates some typical values for the composition of the three different types of rosin:

Components	Gum Rosin	Wood Rosin	Tall Oil Rosin
Pimaric	2%	3%	3%
Palustric	18%	10%	10%
Isopimaric	18%	11%	7%
Abietic	20%	45%	35%

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Dehydroabietic	4%	8%	20%
Neoabietic	18%	7%	4%

These values are just typical values and the actual composition will vary depending on the species of pine tree from which the rosin is obtained and the conditions under which it is processed. However, they illustrate that the general composition of the three rosins is similar.

EPA's comments also highlighted that we may not have adequately described rosin, low boiling fraction (CAS # 68783-82-4) and rosin, distillation overheads (CAS # 68425-08-1), including the manner in which they are manufactured. We indicated in the Test Plan that the two substances are "virtually identical" because the TSCA Inventory descriptions vary by a few words, even though they describe the same substance. The Test Plan also should have stated that for commercial purposes, as well as typical composition, these two substances are the same. These identical low boiling rosin products are produced when rosin is processed at elevated temperatures, but different companies give them different names. Thus, they are duplicate listings in the TSCA Inventory and not different substances. Accordingly, Table 3 of the Test Plan describing the composition of a typical rosin, distillation overheads should have been entitled "Composition of a Typical Rosin, Distillation Overhead or a Typical Rosin, Low Boiling Fraction."

Categorization of Substances / Selection of Test Material

In its Test Plan for Rosin and Rosin Salts, PCA proposed to group six substances and to test rosin to represent the category based on its use as the raw material for all of the other category members, along with having the far greatest production volume. Although EPA believed the grouping of four of the substances was justified by the Test Plan, the Agency questioned whether rosin distillation overheads and rosin low boiling fraction belonged in the category and could be represented by rosin, the representative test substance. The Physicians Committee for Responsible Medicine (PCRM)[1], on the other hand, supported PCA's category as proposed.

The Agency agreed that hydrogenated rosin, rosin potassium salt and rosin sodium salt "are structurally similar or virtually identical to rosin" -- the

[1] PCRM's comments were also submitted on behalf of People for the Ethical Treatment of Animals, the Humane Society of the United States, the Doris Day Animal League, and Earth Island Institute.

representative test substance. However, the Agency was concerned that rosin might not represent rosin distillation overheads and rosin low boiling fraction due to their lower percentage of rosin acids and higher percentages of fatty acids, hydrocarbons and rosin aldehydes, alcohols and esters.

After carefully considering these comments, PCA believes that its category should remain as originally proposed. However, PCA will undertake an additional acute test (OECD 425, the up-down procedure) on rosin distillation overheads (CAS # 68425-08-1) in order to demonstrate that the existing rosin data, as well as the proposed testing of rosin, can represent rosin distillation overheads and rosin low boiling fraction.

Physicochemical and Environmental Fate

EPA agreed that PCA's approaches to all physicochemical and certain environmental fate (water solubility and partition coefficient) was "acceptable for purposes of the HPV Challenge Program." Nonetheless, EPA requested that PCA submit existing data on the components abietic acid, dehydroabietic acid and their corresponding salts. PCA believes that it is impracticable to generate these data on individual resin acids. The substances exist in nature as isomeric mixtures and will easily isomerize if isolated. We also note that our commitment runs to the sponsored chemicals and not their components.

Environmental Fate - Biodegradation

EPA's comments suggest that Table 1 of the Rosin Test Plan indicated that there were adequate data on biodegradation for hydrogenated rosin, but there was no robust summary of this data. However, a review of the Test Plan indicates that Table 1 shows that hydrogenated rosin will be tested for this endpoint; it does not indicate there were adequate data on hydrogenated rosin for this endpoint.

EPA also suggested that PCA should determine whether the robust summary for rosin sodium salt reported the correct test method; we note that the robust summary should have stated that for the rosin sodium salt, biodegradation was determined by the shake culture method. A revised robust summary for this endpoint is attached. EPA also noted a potential discrepancy between the results of biodegradation testing of rosin sodium salt and rosin. We note that this is likely due to the fact that the rosin sodium salt is considerably more soluble than rosin which results in a greater percentage of biodegradation.

In addition, EPA recommended that OECD Guideline 301 should be used for any remaining biodegradation testing. However, because of the differing

solubility of the members of this category, it will be necessary to use two different OECD protocols to test for biodegradation: OECD method 302B will be used for the soluble salts and OECD 301B will be used for the insoluble non-salts.

Ecotoxicity Tests

EPA agreed with the proposed acute toxicity testing of fish, daphnia and algae, but suggested that PCA provide more information on the method as it relates to maximizing solubility, as well as consider conducting a chronic daphnid reproduction test. In contrast, PCRM recommended that PCA forego fish testing in favor of using models like ECOSAR or TETRATOX.

After consideration of these comments, PCA does not intend to amend its Test Plan with regard to the proposed ecotoxicity testing. The methodology for preparing the water for PCA's ecotoxicity testing of rosin is identical to that used to determine the solubility of this substance. This procedure was adopted in order to ensure that ecotoxicity testing was conducted at the limit of actual water solubility. Accordingly, because the solubility of rosin will be empirically determined following stirring for 96 hours, these same conditions will be used to prepare the water samples to be used in conducting the acute fish, daphnia and algae toxicity testing. In addition, as noted in the Test Plan, the effect of both filtering, to further minimize nonspecific physical effects, and of reducing the pH to the lower end of the acceptable range for test organism survival, will also be investigated for changes in toxicological effects. The results of preliminary tests will be used to select the most appropriate test conditions for the definitive test for each species.

We also acknowledge EPA's suggestion that we consider whether daphnid chronic reproductive testing should be undertaken. However, preliminary data suggest that rosin has essentially no aquatic toxicity. It is thus unlikely that chronic testing would be needed. In addition, analytical difficulties could preclude chronic testing in any event.

PCA appreciates EPA's proffer of data regarding abietic and dehydroabietic acids. However, PCA still believes it will be useful to test rosin to represent the mixture as a whole, rather than relying on data for individual components.

Human Health Effects/Acute Toxicity

EPA agreed with PCA's approach as it relates to acute toxicity and repeated dose toxicity. However, the Agency was concerned that the results

from toxicity testing of rosin might not represent rosin distillation overheads and rosin low boiling. Although we have no reason to believe that the compositional differences will affect toxicity, PCA will undertake an additional acute toxicity test (OECD 425) on rosin distillation overheads. The additional test should demonstrate that the proposed rosin testing will be representative of rosin distillation overheads, as well as rosin low boiling.

Human Health Effects/Genotoxicity

EPA disagreed with PCA's reliance on several negative 2-year carcinogenicity studies on rosin to fulfill the genotoxicity endpoint. More specifically, EPA's comments disagreed with the statement from the rosin test plan that "Since the purpose of *in vitro* bacterial and mammalian mutagenicity tests is to determine if a chemical might have the potential to be a direct-acting DNA reactive carcinogen, the negative carcinogenicity studies eliminate the need to test for potential genotoxicity." The comments then go on to list a number of genetic diseases and conditions (e.g., Down's syndrome, cystic fibrosis, hemophilia, sickle-cell anemia, allergies, mental retardation, etc.) with the implication that mutagenicity testing is able to predict the ability of a chemical to cause these adverse outcomes. There is no evidence that the two genotoxicity screening tests that comprise the SIDS battery of tests (i.e., bacterial mutation and chromosomal aberration) have this ability. The likelihood that such testing would predict the non-cancer endpoints noted in EPA's comments is also tempered by the observation in Casarett & Doull's textbook on Toxicology (1996), "No clear evidence exists for the induction of heritable alterations by radiation or chemicals in human germ cells."

In addition, in the early stages of the HPV program, there was uncertainty about the format in which robust summary data would be submitted to EPA. In a meeting with Dr. Oscar Hernandez to discuss this issue, the summarized rosin data were used to illustrate a possible robust summary format. The above statement concerning the use of negative carcinogenicity data to eliminate the need to test for potential genotoxicity was included in the summarized data as part of this discussion. While Dr. Hernandez indicated that mutagenicity testing might indicate the potential for possible endpoints other than cancer, he readily agreed that for purposes of the HPV program, a negative cancer bioassay was a suitable surrogate for genotoxicity testing. Accordingly, bacterial gene mutation and chromosomal aberration testing on rosin will not be undertaken. The headings on the summary tables in the test plan will be changed as suggested to reflect the more accurate designations "gene mutation" and chromosomal aberrations" rather than the bacterial and non-bacterial assays.

Amendment to the Test Plan:

Rosin, distillation overheads (CAS # 68425-08-1) will be tested using OECD 425, the up-down procedure to demonstrate that the results from the rosin toxicity testing are representative of rosin, distillation overheads and rosin, low boiling fraction.

The revised Table 1 below incorporates the additional acute test on rosin, distillation overheads, as well as provides a complete picture of the testing to be performed under this Test Plan.

Chemical and CAS #	Required SIDS Endpoints										
	Partition Coef.	Water Sol.	Biodeg.	Acute Fish	Acute Daph.	Acute Algae	Acute oral	Repeat Dose	Gene Mutation	Chromo-somal aber-rations	Repro/ develop
8050-09-7 Rosin	Test	Test	Adeq.	Test	Test	Test	Adeq.	Adeq.	Adeq.	Adeq.	Adeq Repro./ Test Develop.
65997-06-0, Rosin, hydrogenated	Test	Test	Test	C	C	C	Adeq.	Adeq.	Adeq.	Adeq.	C
68425-08-1, Rosin, distillation overheads	Test	Test	Test	C	C	C	Test	C	C	C	C
68783-82-4 Rosin, low boiling fraction	No test	No test	No test	C	C	C	C	C	C	C	C
61790-50-9, Rosin, potassium salt	Test	Test	Test	C	C	C	C	C	C	C	C
61790-51-0, Rosin, sodium salt	Test	Test	Adeq.	C	C	C	C	C	C	C	C

Table 1
Available Adequate Data and Proposed Testing
On Rosin and Rosin Salts*

Adeq. Indicates adequate existing data
Test Indicates proposed testing
NoTest See test plan; essentially identical to rosin, distillation overheads
C Indicates category read-down from existing or proposed test data on rosin.
* No testing will be conducted for melting point, boiling point, vapor pressure, hydrolysis, photodegradation and transport and distribution between environmental compartments as explained in the test plan.

PCA appreciates the comments from EPA and PCRM, as well as the opportunity to respond. We look forward to sharing the data generated pursuant to the Test Plan.

Respectfully submitted,

Walter L Jones
President & COO

Revised Robust Summary for Rosin, Sodium Salt

ENVIRONMENTAL FATE – BIODEGRADATION	
Test Substance	
Chemical Name	Rosin, sodium salt
CAS #	61790-51-0
Remarks	This substance is referred to as the sodium salt of rosin in the test plan for Rosins and Rosin Salts.
Method	
Method/Guideline followed	Testing was conducted using the Shake Culture method similar to OECD Test Method 301A.
Test Type (aerobic/anaerobic)	Aerobic
GLP (Y/N)	N
Year (Study Performed)	1965
Contact time	32 days
Inoculum	Activated sludge from the Bergen County Sewage Authority treatment plant in Little Ferry, N.J.
Test conditions	Inoculum: Activated sludge from the Bergen County Sewage Authority treatment plant in Little Ferry, N.J. Concentrations of test and reference chemicals: The test and reference chemicals were used at a concentration of 50 ppm. Test Setup: Test medium consisted of magnesium nitrate, calcium nitrate, ferric nitrate, calcium nitrate, cobaltous chloride, diammonium hydrogen phosphate, dipotassium hydrogen phosphate, and monopotassium hydrogen phosphate all dissolved in distilled water. A blank unit (containing all nutrients except the test materials) was treated in the same manner. Microbial cultures were added at a concentration of 10 mg/l on a dry-weight bases to begin the tests. All solutions were placed in Erlenmeyer flasks that were mounted on a shaker for aeration. The study was performed in triplicate. Sampling frequency: Samples were collected for determination of chemical oxygen demand (COD) on an almost daily basis. Controls: Yes. Linear alkylbenzene sulfonate (LAS) Method of calculating chemical oxygen demand: COD was

	calculated as the difference between the measured oxygen concentrations at various sampling times and the start of the test. COD for the samples was calculated by subtracting the COD for the blank controls from the COD in the flasks containing test and reference compounds.
Results	
Degradation % after time	70-80% after 21 days (test article) and 97% after 21 days (reference compound)
Conclusions	These data indicate that the sodium salt of rosin is readily biodegradable.
Data Quality	Reliable with restrictions– Klimisch Code 2e
Reference	Eldib, I.A. 1965. Biodegradability evaluation of (trade name deleted) [rosin, sodium salt]. Eldib Engineering and Research, Newark, N.J.